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Standard Guide for Management of In-Service Phosphate Ester-based Fluids for Steam Turbine Electro-Hydraulic Control (EHC) Systems¹

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INTRODUCTION

The intent of this guide is to provide a summary of management practices for in-service phosphate ester-based fire-resistant fluids used in steam turbine electro-hydraulic control (EHC) applications. Users may also review ISO 11365 and other related publications for additional information (1-5).²

1. Scope

1.1 This guide provides recommendations to achieve safe and reliable operation of EHC systems, recommended levels for required corrective action, and suggested management practices.

1.2 The recommended set of physical and chemical properties of triaryl phosphate esters and their limits have been selected based on past operating experience with various fluids (primarily xylated and butylated triaryl phosphate esters) used in different EHC system designs under different operating and environmental conditions.

1.3 This guide is not intended to replace Original Equipment Manufacturer (OEM) specifications but rather support users experiencing operating problems by recommending limits based on existing knowledge of triaryl phosphate ester degradation mechanisms. This guide should be used outside the warranty period or in cases where no OEM standard exists or is available.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standard-*

ization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

2. Referenced Documents

2.1 ASTM Standards:³

- D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)
- D664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration
- D892 Test Method for Foaming Characteristics of Lubricating Oils
- D974 Test Method for Acid and Base Number by Color-Indicator Titration
- D1169 Test Method for Specific Resistance (Resistivity) of Electrical Insulating Liquids
- D1298 Test Method for Density, Relative Density, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method
- D1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)
- D3427 Test Method for Air Release Properties of Hydrocarbon Based Oils
- D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter
- D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
- D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

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² The boldface numbers in parentheses refer to a list of references at the end of this standard.

³ For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

- D4293** Specification for Phosphate Ester Based Fluids for Turbine Lubrication and Steam Turbine Electro-Hydraulic Control (EHC) Applications
- D4898** Test Method for Insoluble Contamination of Hydraulic Fluids by Gravimetric Analysis
- D5185** Test Method for Multielement Determination of Used and Unused Lubricating Oils and Base Oils by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)
- D6224** Practice for In-Service Monitoring of Lubricating Oil for Auxiliary Power Plant Equipment
- D6304** Test Method for Determination of Water in Petroleum Products, Lubricating Oils, and Additives by Coulometric Karl Fischer Titration
- D6595** Test Method for Determination of Wear Metals and Contaminants in Used Lubricating Oils or Used Hydraulic Fluids by Rotating Disc Electrode Atomic Emission Spectrometry
- D6971** Test Method for Measurement of Hindered Phenolic and Aromatic Amine Antioxidant Content in Non-zinc Turbine Oils by Linear Sweep Voltammetry
- D7042** Test Method for Dynamic Viscosity and Density of Liquids by Stabinger Viscometer (and the Calculation of Kinematic Viscosity)
- D7596** Test Method for Automatic Particle Counting and Particle Shape Classification of Oils Using a Direct Imaging Integrated Tester
- D7669** Guide for Practical Lubricant Condition Data Trend Analysis
- D7690** Practice for Microscopic Characterization of Particles from In-Service Lubricants by Analytical Ferrography
- D7720** Guide for Statistically Evaluating Measurand Alarm Limits when Using Oil Analysis to Monitor Equipment and Oil for Fitness and Contamination
- D7843** Test Method for Measurement of Lubricant Generated Insoluble Color Bodies in In-Service Turbine Oils using Membrane Patch Colorimetry
- D8112** Guide for Obtaining In-Service Samples of Turbine Operation Related Lubricating Fluid
- E659** Test Method for Autoignition Temperature of Chemicals

2.2 ISO Standards:⁴

- ISO 760** Determination of water – Karl Fischer method (General method)
- ISO 2049** Petroleum products – Determination of colour (ASTM scale)
- ISO 3104** Petroleum products – Transparent and opaque liquids – Determination of kinematic viscosity and calculation of dynamic viscosity
- ISO 4405** Hydraulic fluid power – Fluid contamination – Determination of particulate contamination by the gravimetric method
- ISO 4406** Hydraulic fluid power – Fluids – Method for cod-

- ing the level of contamination by solid particles
- ISO 4407** Hydraulic fluid power – Fluid contamination – Determination of particulate contamination by the counting method using an optical microscope
- ISO 6247** Petroleum products – Determination of foaming characteristics of lubricating oils
- ISO 6618** Petroleum products and lubricants – Determination of acid or base number – Colour-indicator titration method
- ISO 6619** Petroleum products and lubricants – Neutralization number – Potentiometric titration method
- ISO 9120** Petroleum and related products – Determination of air-release properties of steam turbine and other oils – Impinger method
- ISO 10050** Lubricants, industrial oils and related products (Class L) – Family T (Turbines) – Specifications of triaryl phosphate ester turbine control fluids (Category ISO-L-TCD)
- ISO 11365** Petroleum and related products – Requirements and guidance for the maintenance of triaryl phosphate ester turbine control fluids
- ISO 11500** Hydraulic fluid power – Determination of the particulate contamination level of a liquid sample by automatic particle counting using the light-extinction principle
- ISO 15597** Petroleum and related products – Determination of chlorine and bromine content – Wavelength-dispersive X-ray fluorescence spectrometry
- ISO 20764** Petroleum and related products – Preparation of a test portion of high-boiling liquids for the determination of water content – Nitrogen purge method
- ISO 20823** Petroleum and related products – Determination of the flammability characteristics of fluids in contact with hot surfaces – Manifold ignition test

2.3 EN Standards:⁵

- EN 14077** Petroleum products – Determination of organic halogen content – Oxidative microcoulometric method

2.4 IEC Standards:⁶

- IEC 60247** Insulating liquids – Measurement of relative permittivity, dielectric dissipation factor (tan d) and d.c. resistivity

2.5 DIN Standards:⁷

- DIN 51794** Testing of mineral oil hydrocarbons – Determining of ignition temperature

3. Terminology

3.1 For definitions of terms used in this standard, refer to Terminology **D4175**.

4. Significance and Use

4.1 This guide outlines the requirements for monitoring the performance of in-service fire-resistant fluids based on triaryl

⁵ Available from European Committee for Standardization (CEN), Avenue Marnix 17, B-1000, Brussels, Belgium, <http://www.cen.eu>.

⁶ Available from International Electrotechnical Commission (IEC), 3, rue de Varembe, 1st floor, P.O. Box 131, CH-1211, Geneva 20, Switzerland, <https://www.iec.ch>.

⁷ Available from Deutsches Institut für Normung e.V.(DIN), Am DIN-Platz, Burggrafenstrasse 6, 10787 Berlin, Germany, <http://www.din.de>.

⁴ Available from International Organization for Standardization (ISO), ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, <http://www.iso.org>.

phosphate esters to accomplish safe and reliable operation of turbine electro-hydraulic control systems.

5. Safety Precaution

5.1 Fire safety tests are used to measure and describe the properties of phosphate esters in response to heat and flame under controlled laboratory conditions and may not accurately represent the hazard of the fluids under actual fire conditions.

5.2 Fires that have occurred in operating turbines have usually been caused by fluid or vapors contacting hot surfaces. For example, fluid that may spill and be absorbed into unprotected thermal insulation can experience an exothermic reaction resulting in a rapid temperature increase. The ignition of the absorbed fluid can occur at temperatures below the fluid autoignition or manifold ignition temperatures.

6. Requirements for Fresh Fluid

6.1 Specification **D4293** provides requirements for the fresh fluid. Alternative, users may check ISO 10050 for requirements.

7. Fluid Deterioration

7.1 Phosphate esters can degrade in several ways. In general, three main degradation processes (that is, hydrolysis, oxidation, thermal degradation) are responsible for the generation of a wide variety of breakdown products.

7.1.1 A common form of phosphate ester degradation is hydrolysis. In the presence of water, aryl phosphate esters tend to break down into their constituent acids, phosphoric acid derivatives and phenols. The hydrolysis reaction is not usually rapid at ambient temperatures but accelerates with increasing temperature and is catalyzed by the presence of stronger acids and some metals.

7.1.2 Another common form of degradation is oxidation. It is a free radical process occurring as the fluid reacts with oxygen. Depending on heat level, oxidation of triaryl phosphate esters occurs via three mechanisms: ‘Low’ temperature (<~200 °C) oxidation produces varnish precursors that eventually precipitate; ‘Moderate-high’ temperature (~300 °C to 1000 °C) oxidation or incomplete combustion occurs as a result of micro-dieseling or wasted energy through internal valve leakage; and, ‘Extremely high’ temperature (>1000 °C) oxidation arises from static discharge (**6, 7**).

7.1.2.1 Varnish is the result of the precipitation/deposition of degradation compounds (also known as varnish precursors) arising primarily from the breakdown of hydrocarbons and phosphates.

7.1.2.2 The mechanism of micro-dieseling involves the rapid adiabatic compression of air bubbles in the system pump. The compression of fluid in the presence of air bubbles leads to an instantaneous temperature increase in the bubble. These high temperatures lead to the formation of incomplete combustion products.

7.1.2.3 The result of excessive internal valve leakage is known to be another thermal degradation mechanism, resulting in fluid darkening and the formation of degradation products.

7.1.2.4 Static discharge, as a result of flow electrification, occurs at extremely high temperatures, resulting in oxidation or

combustion. The rate of charge generation is dependent on a number of factors including flow rate, the electrical properties of the fluid (that is, conductivity), fluid temperature, viscosity, whether the equipment is grounded, the materials of construction and the dimensions of pipework or components through which the fluid is flowing. If the charge on the component is not dissipated, it will accumulate until it is high enough to discharge to a part that is at lower potential, producing a spark.

7.1.3 Thermal breakdown occurs in the absence of oxygen. Depending on the phosphate structure and temperature, different degradation pathways will occur. However, pure thermal breakdown in industrial applications is unlikely as oxygen is normally present in the fluid.

7.2 Attempting to reduce the fluid’s degradation rate will be difficult if the system design, system operating conditions and applied maintenance encourage oxidation to take place or allows moisture ingress. In such cases, the fluid may need to be replaced or subjected to bleed-and-feed more frequently. Alternately, design changes can be made to improve fluid life. Finally, auxiliary equipment may also be used to remove degradation products.

8. Fluid Conditioning System

8.1 To achieve safe and reliable operation of EHC systems, the triaryl phosphate ester hydraulic fluid should be kept clean, with a low water content and acidity. To accomplish this, EHC systems generally have a fluid conditioning system with an acid scavenging filter, operating continually.

8.2 The principle behind the fluid conditioning system is that the rate of acid removal must be greater than its rate of production. If not, control of fluid degradation is lost and acidity can increase quickly because of autocatalytic reactions. In such a scenario, fluid life will be considerably shortened. There is also a greater chance of operational problems. The goal of the conditioning system is not only the removal of acid, but also keeping the fluid dry and clean.

8.3 There are several different types of purification media that have been used in EHC systems.

8.3.1 For many years, fuller’s earth (an attapulugus clay based on aluminosilicates) was used to treat phosphate esters. It allows for removal of active chemical species, thus effectively controlling both fluid acidity and resistivity. It can also adsorb moisture from the fluid, but if other means are not used to keep the system dry, its acid scavenging capacity will be sacrificed. Fuller’s earth’s main disadvantage is that, when exposed to fluids with high acidity, it can release calcium and magnesium. These contaminants can then form metal soaps or salts that may precipitate from the fluid and form sticky gels/deposits in servo-valves, pencil filters, the auxiliary filtration system, and other system components. If an EHC system is exposed to fuller’s earth purification treatment over long periods (for example, several years), users should inspect their reservoirs for gels or deposits, or both. These can occur on the sides as a bathtub-like ring, on the bottom or they may be floating. It is also important that the particulate filter (that is, the ‘polishing filter’) downstream of the acid scavenging filter should have a rating sufficient to prevent fines from getting into

the EHC system (for example, finer than the main pump discharge filter). They can be changed together with the acid scavenging filter, based on OEM guidelines, or experience based on pressure drops.

8.3.2 An alternative adsorbent for low-pressure systems without servo valves and without resistivity limits is activated alumina. It can contain sodium aluminate leading to the release of sodium ions and species containing aluminum hydroxide linkages. These contaminants can interact with acid phosphates contributing to foaming problems. Activated alumina can have a wider particle size distribution than fuller's earth and its hard particles can be abrasive. If used, preference should be given to a low-sodium type.

8.3.3 An alternative option to fuller's earth and activated alumina is a mixture of dried alumina with a Y-zeolite (sodium aluminosilicate). This material is commercially available and is effective at low levels of acidity but less-so at higher levels when compared to fuller's earth. One advantage is that it does not contain either magnesium or calcium, however, sodium is still present. Some fluid suppliers recommend that sodium aluminosilicate adsorbents not be used on fluids previously treated with fuller's earth. In the absence of recommendations from a fluid supplier, users are advised to perform bench tests to verify that fluid properties are not negatively impacted when considering a change from one adsorbent media type to another.

8.3.4 Ion-exchange resins are another purification medium, generally based on a divinylbenzene or polystyrene core, or both, onto which different functional groups have been introduced. These functional groups can react with other chemicals leading either to an exchange of ions or to the adsorption of the chemicals onto their surface. A large number of different ion-exchange resins are commercially available. These resins are mainly used in water-treatment applications. Users are therefore advised to consult with resin providers who have experience in phosphate ester applications. The main advantage of ion-exchange resins is that different resin types can remove stronger or weaker acids, metal soaps and organic varnish. Consequently, a combination of different resin types at different ratios can be used to address specific problems. Resins can also treat significantly degraded triaryl phosphate ester fluids (8). It is important to make sure that the resin or combination of resins used meets the requirements for control of acidity and resistivity.

8.3.5 Long-term application of optimized ion-exchange resins will increase the resistivity of the fluid (9).

8.3.6 Some ion-exchange resins can have as much as 50 % water. In these instances, a separate treatment must be used to address moisture problems. Other ion-exchange resins are available in dried forms to prevent any significant moisture-ingression during their use.

8.4 Users with EHC systems employing fuller's earth, activated alumina, or sodium aluminosilicate, or combinations thereof, should periodically inspect the bottom of their reservoirs for gels or deposits formed from metal soaps. These users are also advised to include metals analysis in their fluid condition monitoring.

9. Flushing the Hydraulic Portion of EHC Systems

9.1 Flushing is another form of corrective action that allows EHC systems to be maintained in safe and reliable condition. There are a multitude of different flushing techniques used to clean EHC systems and this guide will cover only the rinse flush and cleaning agent flush processes. These flushes require a detailed plan, appropriate equipment and an agreed-upon end point. The procedure and equipment required depend on the system design, the extent of any fluid degradation and the desired objectives. It is not possible to have a generic procedure to cover all eventualities. Turbine OEMs may also have a flushing procedure for new systems that can provide a good starting point.

9.2 Rinse Flush:

9.2.1 A system rinse flush may be required when EHC fluid is contaminated with large amounts of mineral oil, which reduces its fire resistance. In addition, it may also be performed when the system is accidentally contaminated with large amounts of water or particles. It may also be performed during a change of fluid type or after major work on the EHC system, or both. A system rinse flush that includes steam valve actuators requires the servo and solenoid valves to be removed and flushing valves installed in their place. If the steam valve actuators are removed during a system rinse flush, then flushing jumpers can be installed at the fluid piping. Emergency trip circuits are flushed using flushing valves or jumpers. Accumulators must be exercised multiple times to remove foreign material or degradation by-products that may be trapped. System pumps can be used to perform this process if suction strainers are used to protect the EHC pumps. Suction strainers must be checked regularly. To increase the flow velocity using the system pumps, both pumps are generally used. Before using the system pumps to perform a flush, it is extremely important to identify the type and level of contamination in the system. If foreign material enters the EHC system pump suction, catastrophic failure can occur and additional metal contamination will be introduced through the case drain line. This applies to pressure compensated pumps. External flushing units ensure that system pumps are protected. The rinse flush process may be performed with the operating fluid as long as the fluid condition is in specification, with no history of high acid number, water and low resistivity values. Alternatively, specific rinse flushing fluid may be provided by the fluid supplier.

9.2.2 Steps of Rinse Flush:

9.2.2.1 Draining and inspecting the reservoir.

9.2.2.2 Installing flushing valves in place of servo/solenoid valves.

9.2.2.3 Refilling with flushing fluid.

9.2.2.4 Cycling actuators and accumulators.

9.2.2.5 Continuous fluid sampling. An in-line or on-site particle counter can be helpful.

9.2.2.6 Draining and inspecting the reservoir after flushing.

9.2.2.7 Replacing all system filters.

9.2.2.8 Refilling the EHC reservoir with new fluid that has been verified to be in specification.

9.2.2.9 System start up and verifying EHC system operation and fluid condition. Typically, samples are taken between 72 h to 96 h after fluid recirculation.

9.3 *Cleaning Agent Flush:*

9.3.1 A system cleaning agent flush should be performed when users need to remove gel/varnish or heavy soot from thermal degradation found in the system. These deposits can form when a high acid number is not corrected for long periods (for example, more than three months). Reservoir inspections can be done to identify these deposits or an MPC test may help to determine the amount of suspended varnish or soot in the fluid. The right cleaning agent flush process can dissolve deposits and put them in solution, allowing them to be removed from the reservoir. This process should be performed by a professional that has extensive knowledge of EHC system operation and experience with that specific cleaning agent. External flushing units should be used to perform this process due to the high amount of contamination generated during the flush. EHC system pumps have a greater chance of failing if used to perform this process. This process requires removing all servo and solenoid valves from the system, and installing flushing valves on the actuators. Emergency trip solenoids are removed and flushing valves are installed on the trip manifold. Accumulators are removed from the system and disassembled to inspect the piston/bladder for contamination, then reassembled with new seal kits or bladders, or both. It is very important to use compatible elastomers. After the cleaning agent flush process is complete, it is necessary to perform the rinse flush process to remove the cleaning agent fluid, and contamination generated from this process.

9.3.2 *Steps of Cleaning Agent Flush:*

9.3.2.1 Draining and inspecting the reservoir.

9.3.2.2 Installing flushing valves in place of servo/solenoid valves.

9.3.2.3 Refilling the system with original fluid and cleaning agent.

9.3.2.4 Cycling actuators.

9.3.2.5 Removing and inspecting accumulators, and rebuilding them if required.

9.3.2.6 Continuous fluid sampling. An in-line or on-site particle counter can be helpful.

9.3.2.7 Draining fluid at all low points, including heat exchangers, and inspecting the reservoir after the cleaning agent flush process.

9.3.2.8 Refilling the EHC reservoir with rinse flush fluid.

9.3.2.9 Performing the rinse flush process.

9.3.2.10 Continuous fluid sampling until fluid samples exceed OEM or user specifications.

9.3.2.11 Draining fluid at all low points.

9.3.2.12 Cleaning the reservoir.

9.3.2.13 Replacing all system filters.

9.3.2.14 Reinstalling accumulators and recharging with nitrogen.

9.3.2.15 Refilling the EHC reservoir with new fluid.

9.3.2.16 System start up and verifying EHC system operation and fluid condition. Typically, samples are taken between 72 h to 96 h after fluid recirculation.

10. Condition Monitoring Program

10.1 The principle behind the fluid conditioning system is that the rate of acid, moisture, and particle-removal must be greater than the rate of their production or ingress. There are, however, occasions where the fluid may deteriorate at faster rates due to changes in operation, environment, or maintenance, or combinations thereof. Also, from an economic point of view, the fluid may reach a stage where its replacement is the preferable option. Therefore, to avoid uncontrolled fluid deterioration and maintain the safe and reliable operation of EHC systems, users must implement a condition monitoring program to assess fluid condition and implement appropriate corrective actions.

10.2 Due to the scope of this guide, only the most critical physical and chemical fluid properties will be discussed. Other materials are available such as from some turbine OEMs, EPRI, and VGB (1-5).

10.3 It is good practice to filter fluid when transferring from drums into the turbine EHC system. The rating of the filter element should be as good or better than the system filter element. In extreme cases, it may also be necessary to dry the fluid before operation. Note that triaryl phosphate ester fluids have a low viscosity index and become more viscous at low temperatures. Consequently, fluid in the drums should be allowed to warm to about 20 °C or higher. The actual temperature will depend on the equipment used. Having a warm fluid (that is, at lower viscosity) is very important in case the transferring unit filters have a pressure bypass valve. In such a case, cold fluid could enter the system unfiltered.

11. Fluid Sampling

11.1 Sampling of in-service phosphate ester fluids should be in accordance with Guide D8112.

11.2 If a fire-resistant fluid sample is required from a drum, then the sample should be represented by an “all-levels sample” obtained by tube sampling. Additional information on sampling is available in Practice D4057.

11.3 Care should be taken to ensure that the fluid is mixed properly before samples are drawn and that the samples are representative of the whole charge.

11.4 Sampling intervals should be specifically set for different EHC systems depending on their criticality and the severity of their operating conditions. Users are advised to refer to OEM instructions or other regulatory guidelines for guidance in selecting appropriate sampling intervals. In the absence of specific directions, users are advised to take samples as follows:

11.4.1 Upon receipt of a new fluid per Specification D4293.

11.4.2 After a new fluid charge has been in circulation for at least 24 h. Typically, samples are also taken between 72 h to 96 h after fluid recirculation.

11.4.3 On a monthly basis for testing as outlined in Table 1.

11.4.4 On an annual basis for testing as outlined in Table 1.

11.4.5 Prior to outage or significant maintenance action.

11.4.6 After a fluid has been returned to circulation (preferably for 72 h to 96 h) following an outage or significant maintenance action.

TABLE 1 Suggested Tests and Frequency of Routine Condition Monitoring Program for In-Service Triaryl Phosphate Ester Fluids Used in EHC Systems

Property / Frequency	Test Method	Alternative Method	Monthly	Annually ^A	As Required (Troubleshooting)
Appearance	See 12.1	ISO 2049	x	x	
ASTM Color	D1500		x	x	
Acid Number	D664 or D974	ISO 6619 or ISO 6618	x	x	
Water Content	D6304	ISO 760 ISO 20764	x	x	
Volume Resistivity ^B	D1169	IEC 60247	x	x	
Fluid Cleanliness	ISO 11500 ISO 4406	ISO 4405 ISO 4407 D7596	x	x	
Metal Content	D4898 D6595		x	x	
Membrane Patch Colorimetry (MPC)	D7843		x	x	
Linear Sweep Voltammetry	D6971		x	x	
Viscosity	D445	ISO 3104 D7042		x	
Chlorine Content ^B	See 12.12	EN 14077 ISO 15597		x	
Air Release	D3427	ISO 9120		x	x
Foaming	D892	ISO 6247		x	x
Mineral Oil Content	See 12.8			x	x
Manifold Ignition Temperature ^C	See 12.15	ISO 20823			x
Auto-Ignition Temperature ^C	E659	DIN 51794			x

^A Annual tests can be deferred but for trending or to get a benchmark for troubleshooting, or both, they should be run at least every few years or prior to and following a scheduled major outage, or both.

^B For lower pressure systems without servo valves, resistivity and chlorine testing are not typically necessary.

^C Manifold Ignition and Auto-Ignition tests are usually performed after specific concerns are identified.

12. Recommended Practices for Fluid Condition Monitoring

12.1 Appearance:

12.1.1 An appearance check of fluid samples is an important screening test and an initial indicator of fluid and system conditions. It allows users to identify if any significant change has occurred since the last sampling. Examples include fluid clarity (for example, milky or hazy appearance due to suspended water, presence of air bubbles, mineral oil contamination greater than the fluid solubility limit, significant contamination with fine particles, etc.), darker than normal color, free water layer, and sediment. Users may also assess whether the fluid has a burnt or unusual odor.

12.1.2 Fluid samples collected in clear, colorless glass bottles are preferable. Samples in transparent plastic bottles also allow for visual assessment of fluid condition. In either case, the sample can be slowly inverted and the bottom checked for sediment. If there is a free water or mineral oil layer, it should be at the top of the fluid sample.

12.1.3 It is a good practice to repeat the appearance check after letting the samples sit overnight in a closed container at room temperature or after 24 h in an open container. This will allow entrained gases to escape, minimizing confusion between air and water contamination. This also allows some particles to settle.

12.1.4 To identify abnormal fluid, users should be familiar with the appearance of new fluid. Therefore, an unused sample of the fluid in current use should be retained and available for comparison. The unused fluid sample should be kept in a cabinet or light-proof container to prevent darkening because of UV from lights.

12.2 ASTM Color:

12.2.1 Some darkening of triaryl phosphate esters can be normal as a result of oxidation, micro-dieseling or thermal degradation, or both.

12.2.2 Color is typically determined by Test Method D1500 with a range from 0.5 (for example, colorless) to 8 (for example, almost black). Some samples may be too dark or may not match color standards. In this case, they are reported as “Does Not Match Standard Colors”. However, additional comments should be provided to describe the color observed. Photographs can be helpful.

12.2.3 In-service fluid will slowly darken but, typically, the darkening should level out after a few years, unless abnormal conditions are present.

12.2.4 If rapid or unusual darkening is detected, then users should investigate the root cause (for example, hot spots in the EHC system, micro-dieseling, a change in operating conditions such as running two pumps at the same time, low fluid level in the reservoir, lower make-up rate, contamination, steam lines too close to the fluid line, deteriorated insulation, etc.). An infrared camera may assist in the detection of hot spots in the EHC system.

12.2.5 After identifying the root cause of the problem, corrective actions could include bleeding off a portion of the in-service fluid. Online treatment might also be possible but this would depend on the cause of fluid darkening. Such treatment could include the use of ion-exchange resins, activated carbon, or the use of electrostatic filtration.

12.3 Acid Number:

12.3.1 This property is the most important parameter used for monitoring EHC fluid condition.

12.3.2 As discussed in Section 7, Fluid Deterioration, acidic compounds are a result of hydrolysis, oxidation, or thermal

breakdown, or combinations thereof. The acids that form may be stronger or weaker. Stronger acids are generally arylphosphoric acids that form following hydrolytic phosphate ester breakdown. Weaker acids may be phenols that arise from hydrolysis or organic acids (for example, carboxylic acids) that arise from oxidative breakdown of alkylphenols. Stronger acids are more reactive than their weaker counterparts and, therefore, tend to be more harmful. The concern with high acidity is that the degradation process is auto-catalytic, significantly increasing the rate of fluid deterioration. In addition, if solid media such as fuller's earth, activated alumina, or an alumina/zeolite blend are used in the purification system, high acidity can form soluble metal soaps or salts. These contaminants may promote foaming when in solution, or impair valve operation if precipitated. If acid number gets too high, these adsorbent media may be less efficient.

12.3.3 If weaker acids (for example, alkylphenols) are not removed, high MPC varnish potentials may result due to oxidation or thermal degradation, or both.

12.3.4 It is important to understand the root cause of acid increase and to eliminate the source.

12.3.5 Depending on their acid number, users may consider the application of a different purification medium. While stronger acids are more harmful, their higher relative reactivity tends to make them easier to remove. Weaker acids, on the other hand, may be more difficult to remove and can accumulate if an appropriate purification medium is not employed. It is therefore important to check the level of weaker acids present. In extreme conditions, corrective actions may require draining a portion of the in-service fluid and replacing it with new fluid.

12.3.6 Current operating experience with triaryl phosphate ester fluids in EHC systems suggests that maintaining the acid number below 0.10 mg KOH/g will allow users to operate in sustainable, good condition. It may be difficult to maintain the acid number at this level with some EHC system designs. In such cases, additional purification treatments or system modifications to reduce operational severity should be considered.

12.3.7 Operating experience also indicates that acid number will increase more rapidly once its level exceeds 0.20 mg KOH/g. Acid number increases tend to be especially rapid when stronger acids are present. The rate of hydrolysis for butylated phenyl phosphate esters is generally higher as compared to xylyl phosphate esters. Therefore, systems containing butylated phenyl phosphate ester fluids may require more frequent replacement of acid scavenging filters as compared to systems containing xylyl phosphate ester fluids under similar conditions.

12.3.8 There are two standards for determination of fluid acidity. Test Method **D664**, the potentiometric method, measures total acid number from both stronger and weaker acids, and can distinguish between the two. Test Method **D974**, the colorimetric method, partially measures the stronger acids and does not measure the weaker acids. For dark in-service fluid, the potentiometric method **D664** is preferred because of difficulty in identifying a definite color change for the colorimetric **D974** test method. The colorimetric method does not

allow for the determination of acid strength. For trending purposes, it is recommended that results be obtained from the same method.

12.4 *Water Content:*

12.4.1 Water contamination causes the slow breakdown of triaryl phosphate esters by hydrolysis, producing acid phosphates (stronger acids) and phenols (weaker acids). A high concentration of acid phosphates in operating fluid catalyzes the fluid degradation process. Also, the rate of hydrolysis will increase with water content and with the temperature of the fluid.

12.4.1.1 Water contamination is typically the result of ingress from the environment since phosphate esters are hygroscopic and can absorb over 1000 mg/kg of water. Note that desiccant breathers may not be adequate to maintain desired water levels since hygroscopic EHC fluids may pull moisture from breathers.

12.4.1.2 Water may also elute from some acid adsorbent media. Many commercially available ion-exchange resins have a significant moisture content (generally around 50 %) and the use of a wet resin can lead to significant water ingress. Resin providers with experience in lubricant applications will often provide dried ion-exchange resins which eliminate or mitigate this risk. Alternatively, some resin suppliers recommend the use of various drying technologies alongside wet resins (for example, reservoir dry air purge gases or vacuum dehydration). Users are advised to consult with resin suppliers who are experienced in EHC applications to determine whether their resins may contribute water and, if they do, what steps should be taken to mitigate the associated risks.

12.4.1.3 Occasionally, cooler leaks may also be a source of water ingress.

12.4.2 If there is no evidence of abnormal water ingress, then a vacuum dehydration unit or reservoir dry gas purge can be the quickest way to dry the fluid and manage its moisture content. However, it is recommended that the fluid should not be over-dried (below 300 mg/kg) to avoid negative impacts on fluid condition.

12.4.3 Preferably, the fluid moisture level should be maintained in the range of 300 mg/kg to 500 mg/kg. Typically, water content of 1000 mg/kg is considered as the turbine OEM maximum allowable level. Trend plots can help to identify the source of water ingress and the most suitable corrective action. Seasonal variations in atmospheric humidity also influence the fluid water content, with lower levels normally being found in drier winter months.

12.4.4 If, due to contamination, a significant free water layer is formed, corrective actions may include pumping or siphoning (or other water-removal techniques) of the water floating on the surface or in the worst-case scenario, total fluid replacement.

12.4.5 If water contamination due to external ingress is a continuous problem, users should consider installing a vacuum dehydration unit, a dry gas purge system or a membrane dryer.

12.5 *Volume Resistivity:*

12.5.1 EHC systems with servo valves might experience servo valve spool corrosion due to electro-kinetic wear. While volume resistivity is not the only factor associated with this